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Indian J. Phys. **51A**, 142–144, (1977)

Study of island growth in vapour deposited lead sulphide films by optical diffraction from the electron micrograph.

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(*Received* 27 *January* 1976. *revised* 14 *June* 1976 *and* 13 *September* 1976)

Study of the properties of vapour deposited lead sulphide films is of importance because of their applications in devices as infra-red detectors. Since most of the magnetic, electrical and optical properties of polycrystalline films depend critically on the thickness and microstructure, a study of the structural characteristics of the films would be highly useful. The use of small angle X-ray scattering and small angle electron diffraction for determining the particle sizes and size-distributions is well known (Guinier & Fournet 1955, Ferrier 1969). Beeston *et al* (1972) have reviewed the various studies on the optical diffraction from electron micrographs and have emphasised its uses in obtaining structural information. Hence in the present investigation, the island sizes have been determined by the optical diffraction from the electron micrographs and also by the conventional technique. Preferred orientation and the sizes of the coherently diffracting crystallites have been determined by X-ray line broadening method.

Thin films of different thickness were deposited on glass substrates kept at room temperature using an Edwards Vacuum coating unit under a vacuum of 10^{-5} torr. The thickness of the films was estimated by the loss of mass method. The surface structures of the PbS films used in the present investigation were studied by the carbon replica technique. For obtaining better contrast the

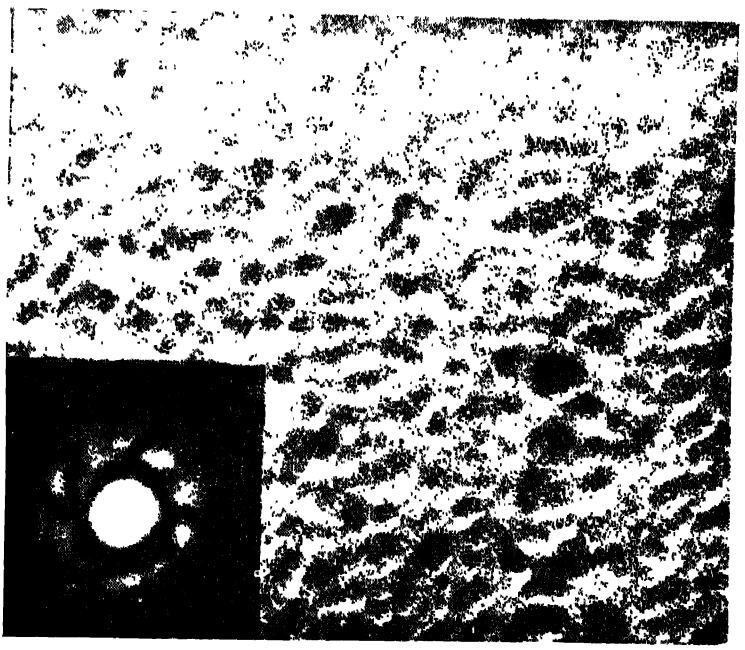

Fig 1. Electron micrograph of 2350 A thick PbS film and its optical diffraction (in the insert).

replicas were shadowed with an alloy of gold and palladium. The micrographs were obtained with the help of an A.E.I. EM 6 electron microscope. The optical diffraction patterns were obtained with a laser diffractometer constructed in the laboratory using 1 mW Spectra Physics He-Ne laser ($\lambda = 6328$ A.U.) as the source. It is similar to the one described by Beeston *et al* (1972). A typical electron micrograph obtained in the present investigation has been shown in figure 1 with its optical transform in the inset. The intensities were obtained from the photographs with the help of a recording microphotometer.

From Debye-Raleigh theory of scattering of light by spherical particles, the relative intensity of scattered light at an angle θ is given by (Debye 1947)

$$I_\theta = \left[\frac{3}{U^3} (\text{Sin } U - U \text{ Cos } U) \right]^2 \quad \ldots (1)$$

where

$$U = \frac{2D}{\lambda} \text{ Sin } \left(\frac{\theta}{2} \right) \quad \ldots (2)$$

where D is the diameter of the scattering particles, λ is the wavelength of the light and θ is the scattering angle. Eq. (1) is valid for the case of $m \to 1$, where m is the ratio of the refractive index of the scattering particles and that of the medium. Eq. (1) has first minimum and maximum of intensity at $U = 4{\cdot}4924$ and $U = 5{\cdot}7617$ radians respectively. For other values of m, the corresponding value of U for different minima and maxima can be obtained from Mie theory calculations (Maron *et al* 1963). The angular positions of the minima and maxima are given by

$$\frac{D}{\lambda m} \sin \left(\frac{\theta_i}{2} \right) = k_i \text{ or } K_i \quad \ldots (3)$$

where k_i or K_i correspond to ith minima and ith maxima respectively. Values of k_i and K_i have been calculated by many workers for different cases (Maron *et al* 1963, Nakagaki & Heller 1960 and Kerker *et al* 1964). The above method has been applied by Dandliker (1950), Dezelic & Kratohvil (1961), Maron & Elder (1963) and others for determination of particle size and the results are in good agreement with the electron microscopic data.

In the present investigation the island sizes were measured from the positions of the maxima and minima of the scattered intensity and by using eq. (3). The island sizes were also measured from the electron micrographs by the usual method.

X-ray diffraction profiles of the films were recorded with the help of a Norelco X-ray diffractometer and the crystallite sizes were obtained from the line profiles by using the method of integral width (Williamson & Hall 1953).

It is observed that in initial stages of deposition the islands show an irregular structure. These islands however become almost circular in shape for films of

greater thiskness. It is also observed that initially the increase in island size with the thickness is very slow. However for greater thicknesses the rate of increase in the island size is much greater. The optical diffraction patterns for most of the micrographs show broad circular inrerference maxima, thus indicating dense packing and uniform shape of the islands. Typical results for some of the films have been shown in table 1.

Table 1. Structural parameters of lead sulphide films.

Thickness in A U	Island size from electron micrographs in (A U)		$\frac{I_{200}}{I_{111}}$	Particle size from X-ray analysis in A U	
	Conventional technique	Optical diffraction technique		(111)	(200)
1500	200	300	—	—	—
1750	250	300	·57	300	100
2350	200	200	·60	350	120
4500	1000	1100	·64	600	300

It is clear from table 1 that at the initial stages of the growth there is an increase in the number of the islands with little increase in their size. Some times even there is an increase in the number of islands at the cost of the size. X-ray analysis of the various films show that the ratio of the intensities I_{200}/I_{111} is of the order of 0·6, thus indicating a preferential growth of the crystallites with 111 planes parallel to the substrate. Measurement of the crystallite size perpendicular to the 111 and 200 planes shows that the crystallite size in the 111 direction is larger than that in the 200 direction. It is also observed that the anisotropy in the crystallite size decreases with the increase in thickness.

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